In-line Quality Control of PEM Materials

6/6/16 – 6/10/16

DOE Annual Merit Review, Washington D.C.

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Project ID #: MN016
Contract No.: DE-SC0013774
PM: Nancy Garland

Overview

Timeline and Budget

SBIR Phase I

- June 2015 – March 2016
- $150,000
  - Total Project: $150,000
  - Total recipient share: $0
  - Total DOE funds spent: $141,118

Technical Targets

Build a prototype system to simultaneously measure:

- Defects in a moving membrane web
- Membrane thickness over the full web width

Barriers Addressed

- **E. Lack of Improved Methods of Final Inspection of MEAs**
- **H. Low Levels of Quality Control**

Partners/Collaborators

- National Renewable Energy Laboratory: Mike Ulsh, Peter Rupnowski

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Relevance

- **<u>DOE Objectives</u>: Improved quality control to improve reliability and reduce automotive fuel cell stack costs to $20/kW by 2020 at 500,000 units/year**

- **<u>DOE Targets</u>**
  - Develop in-line diagnostics for component quality control and validate performance in-line
  - Increasing the uniformity and repeatability of fabrication
  - Reduce labor costs and improve reproducibility by increasing automation
  - Identify cost drivers of manufacturing processes

- **<u>Mainstream Engineering Targets</u>**
  - Demonstrate real time automated in-line defect and thickness mapping on NREL web line
  - Improve manufacturing process by providing real time feedback on quality metrics
  - Scan the membrane with 100% coverage, marking and logging defective regions

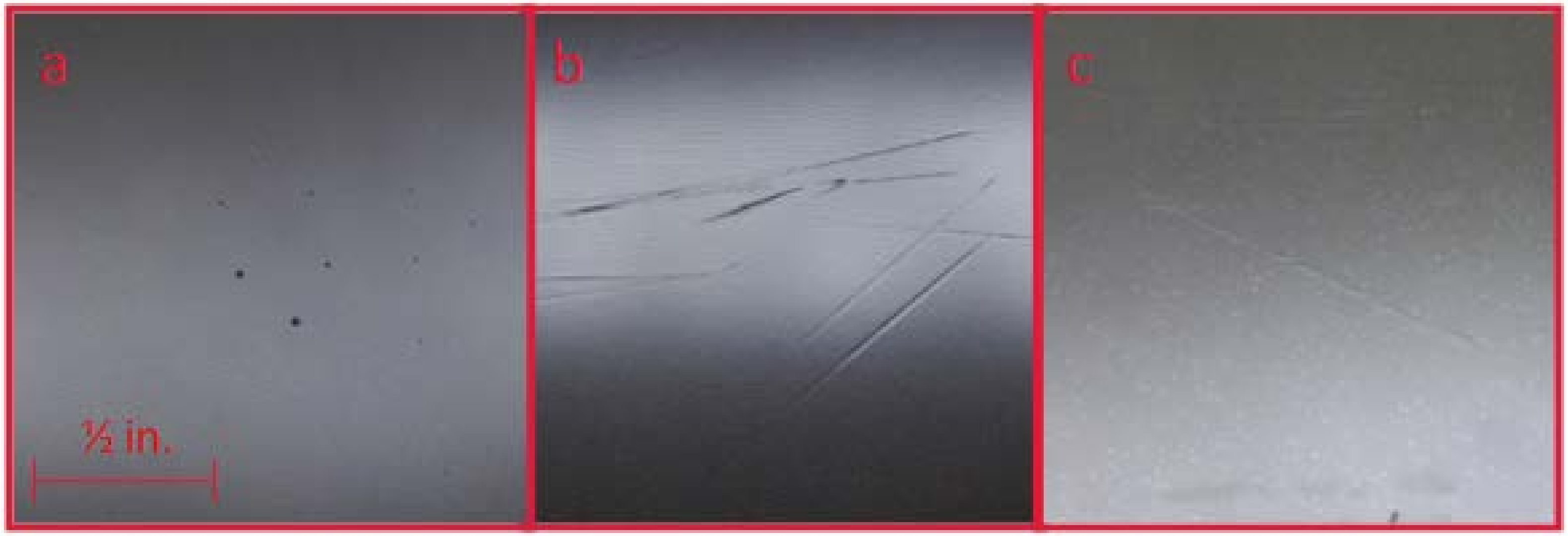
In-line QC of PEM Materials

- Demonstrate membrane defect detection using in-line machine vision optical techniques
- Develop membrane thickness mapping capable of real time measurement across the full web
- Determine membrane rejection criteria
- Develop software to automate analysis, defect logging and real time identification of critical defects
- Fabricate and test a prototype incorporating an optical sensor system
- Apply methods to an array of membrane materials at web speeds up to 100 ft/min

Membrane Defect Types

- **Examined three primary types of defects**

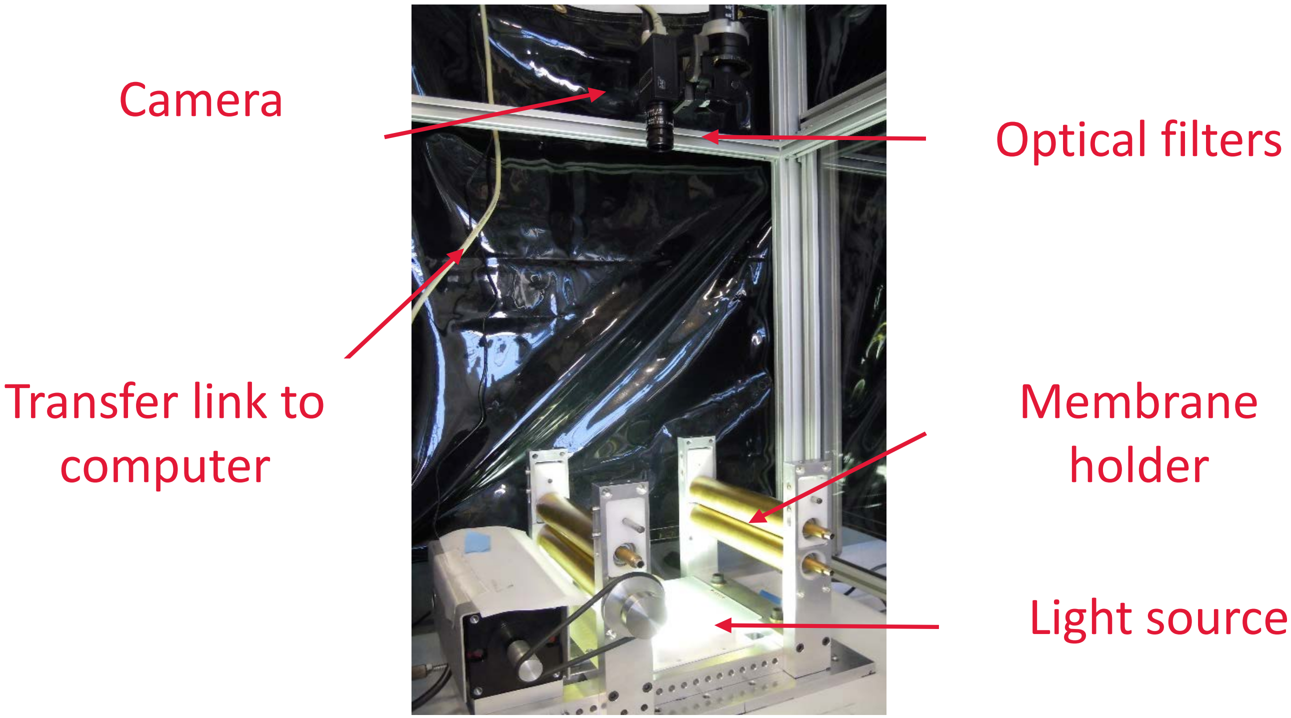
Pinholes **Scratches** **Folds**



Images taken with edge-lit compact camera

Static Measurements

- **Determination of thickness and defect detection limits for the current optical hardware**



- **Mainstream's cross-polarized near-UV-Vis optical arrangement improves the defect resolution**

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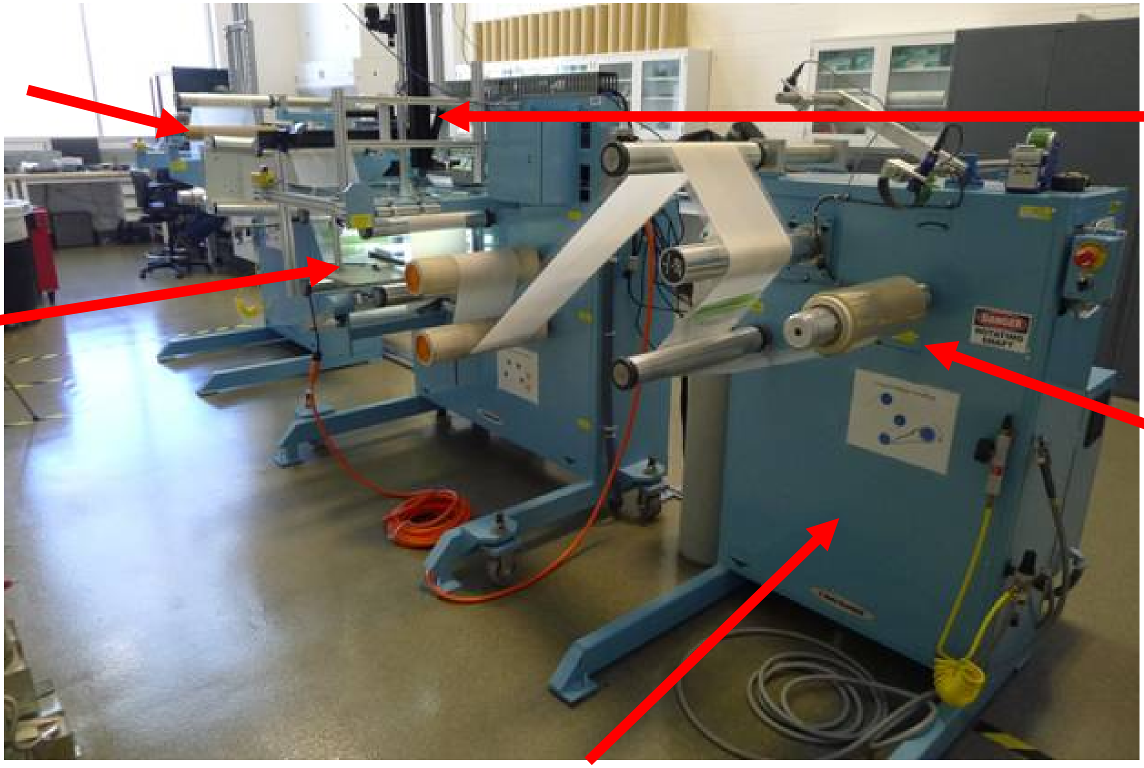
Moving Web Line Measurements

Mainstream's system tested on NREL's web line up to 100 ft/min

Rewind Station with web steering

Mainstream's in-line optical diagnostics

Light source and filters



Membrane web with tension control

Unwind Station

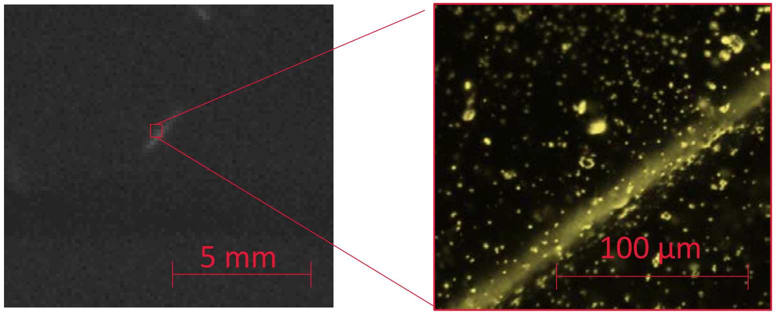
Milestones

Phase I Milestones	Phase I Result
Identify the smallest discernible defect size and characteristics for PFSA membranes	▪ Unsupported membranes ▫ 25 μm diameter pinhole ▫ 10 μm width scratch ▫ 100 μm width fold or crease ▪ Supported membranes ▫ 100 μm diameter pinhole ▫ 100 μm width scratch ▫ 100 μm width fold or crease
Determine membrane thickness to ±1 μm for a 25 μm thick membrane	▪ Nafion®-115: ±1 μm for 132 μm film by polarimetry ▪ Nafion®-211: ±0.5 μm for 25 μm film by absorption
Demonstrate defect and thickness analysis in real-time up to 60 ft/min	Demonstrated at up to 30 ft/min for Nafion®-211 with real-time processing; 100 ft/min with image post-processing
Develop membrane defect criteria and identify defects on a moving web	Found 100% of 100 μm pinholes in Nafion®-211 at 30 ft/min in real-time; 100 ft/min with post-processing
Integrate an encoder and printer to mark defects locations in real time	Marked 35-of-35 defects in real-time. Printed every 1 foot for 50 feet at variable web speed from 1 to 60 ft/min.

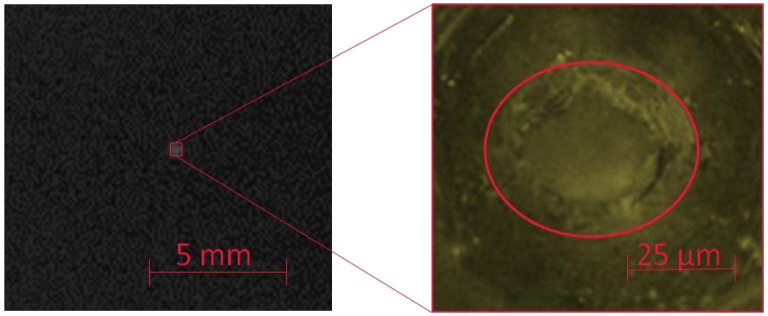
Optical arrangement provides a significant improvement in the defect resolution for a given camera pixel count

Defect Limit-of-Detection

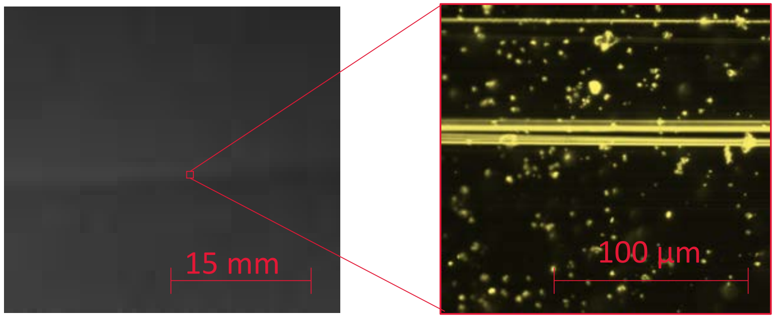
- **Smallest detectable defects where the left image is from the Mainstream's detector and the right is from a high-powered optical microscope**



Fold defect in Nafion®-211 at 100 µm width by 500 µm length



Pinhole defect in Nafion®-211 at 25 µm



Scratch defect in Nafion®-211 at 10 µm width by 100 µm

Membrane Thickness Mapping

- **High resolution thickness mapping by polarimetry across the membrane web**

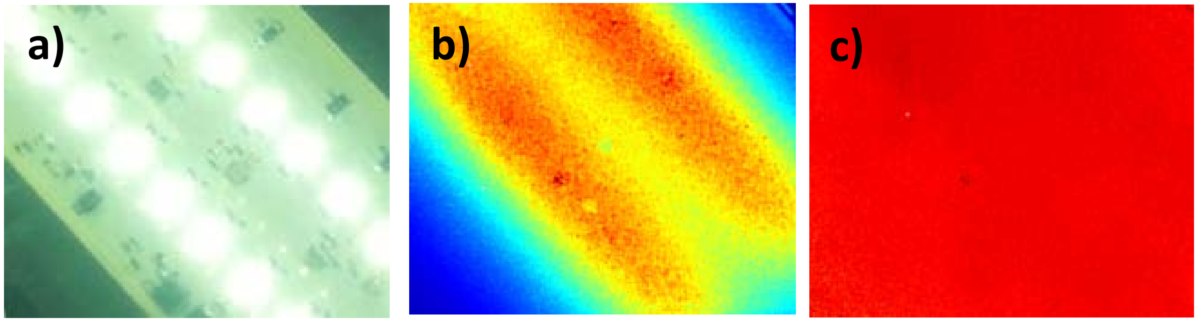
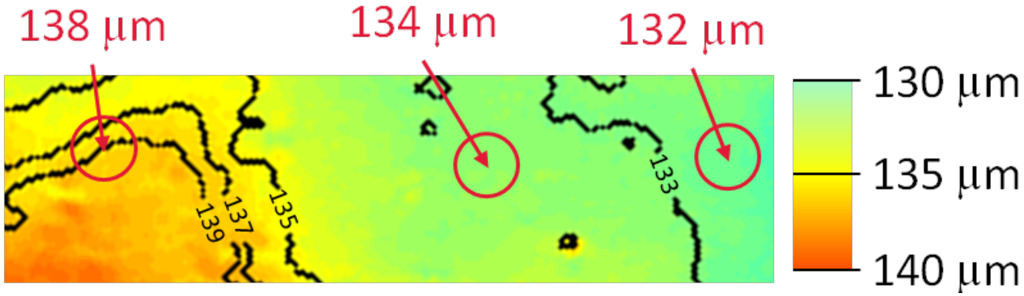


Image of Nafion®-115: (a) regular backlit photograph, (b) colorized image from Phase I area-scan camera, (c) image with background compensation



Thickness Map of a deformed Nafion®-115 sample, where the red circles are micrometer measurements

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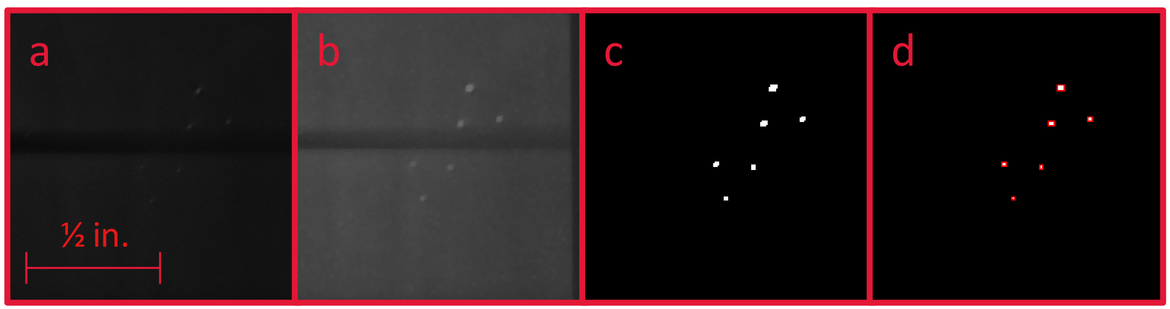
Image Analysis

- **Custom software and optical enhancement provides improved defect resolution**

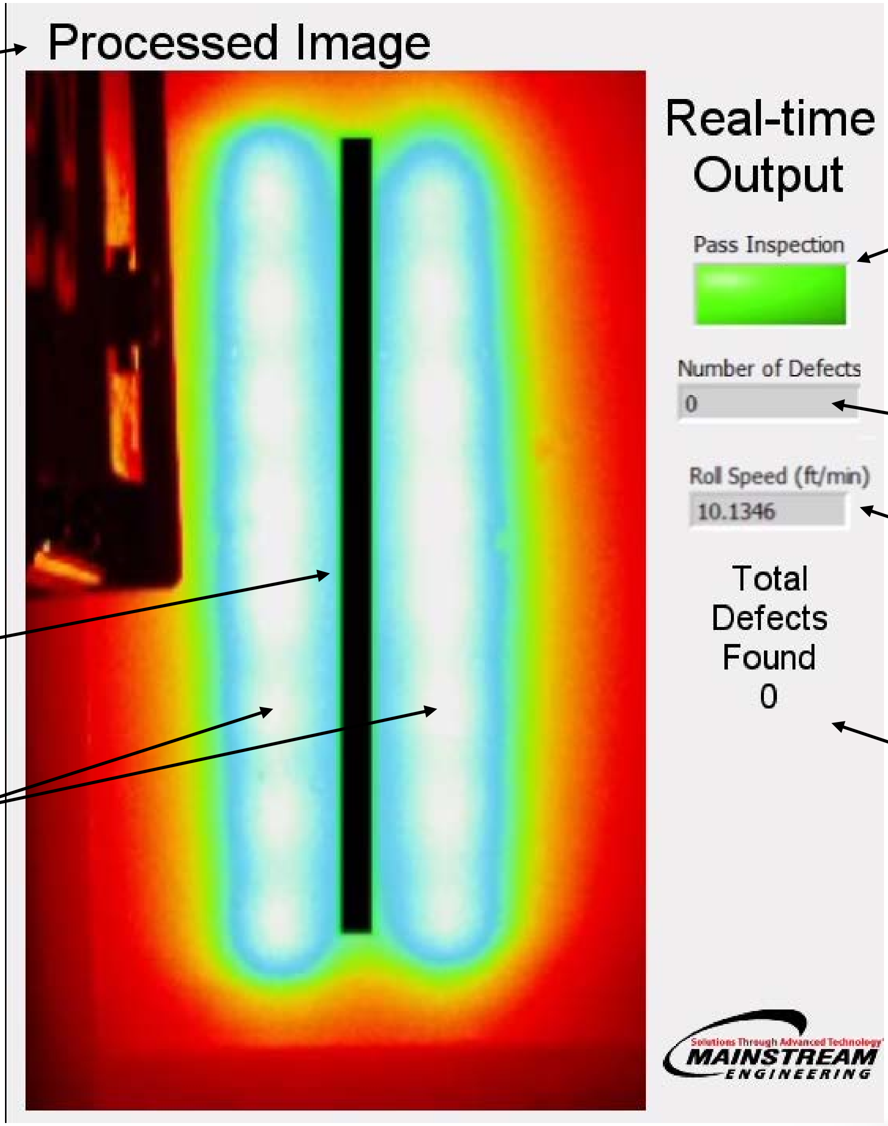
	a	b	c	d
The software process	Image acquisition and transfer from camera to computer	Image enhancement effects	Image conversion to binary image	Defect detection and logging

Resultant image



Prototype Image Analysis UI

Monochrome image represented as a color gradient with defects marked in the black region



Active analysis region

Uniform Light Source

Inspection status – green indicates no defects present

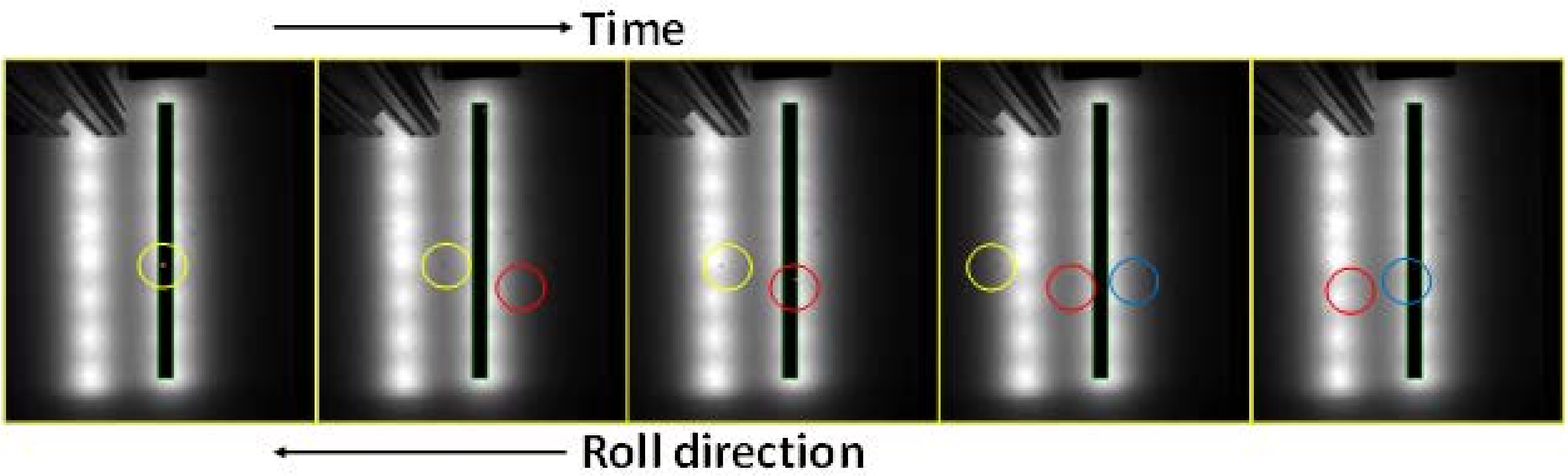
Number of defects found in active analysis region

Line roll speed for determining necessary camera parameters and marking defects

Total of defects found since start of the roll

Defect Detection up to 100 ft/min

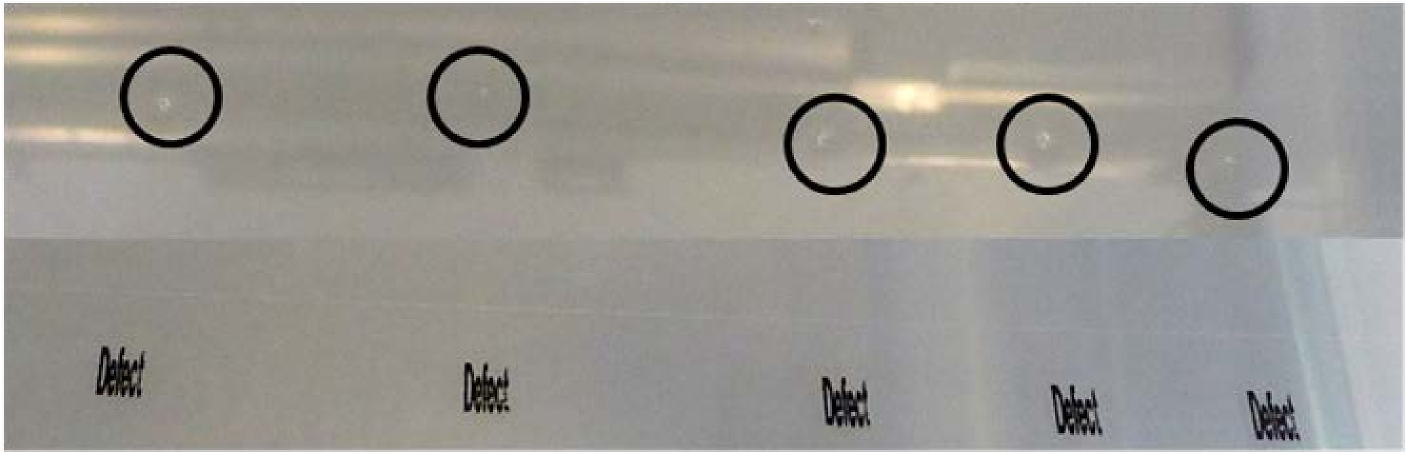
- **Defects accurately detected in a range of supported and unsupported PEM membranes**
- **40/40 100 µm and 40/40 500 µm pinhole defects identified**
  - In real-time up to 30 ft/min, with post-processing up to 100 ft/min



Stop-frame time series of images showing roll-to-roll defect detection in Nafion®-211 at 30 ft/min

Defect Location Printing

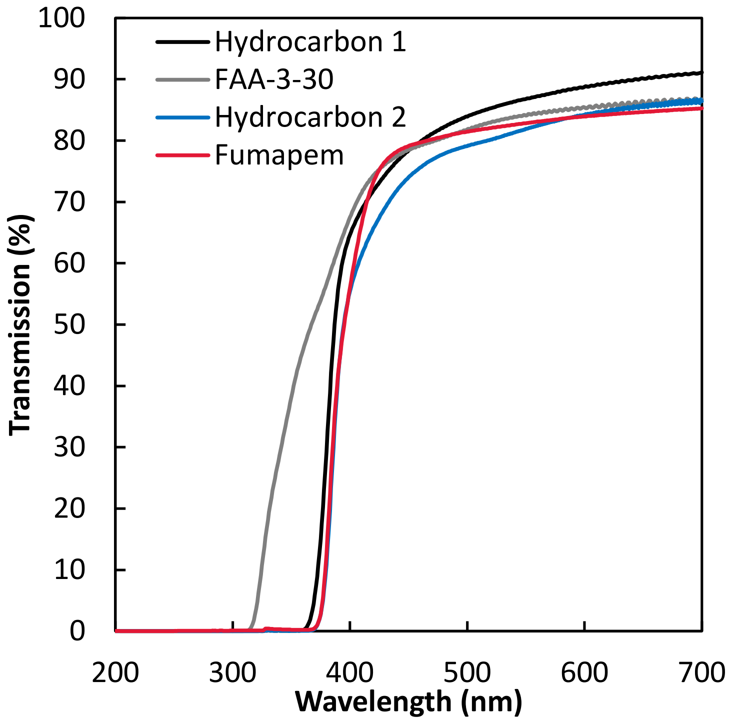
- **PET defects detected at 10 ft/min marked by the printer**
- **Encoder used to measure roll speed and determine printer timing**

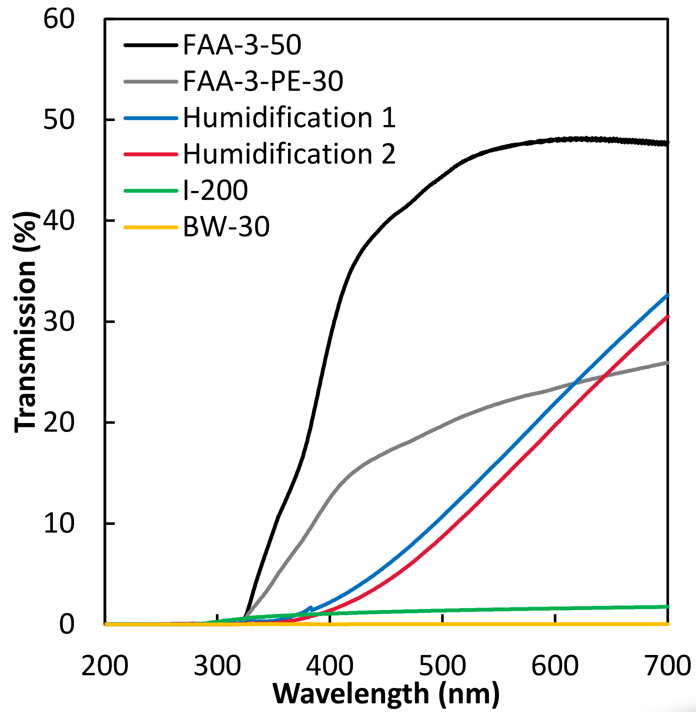


Five 500 µm pinhole PET defects, highlighted with black circles, automatically detected and marked by Mainstream's setup at 10 ft/min

Other Membrane Applications

- Alternative membranes for reverse osmosis, anion exchange, hydrocarbon PEM, and electrolysis
- All transmit over 10% in the UV/Vis except for I-200 (AEM) and BW-30 (reverse osmosis)





Collaborations

Institution	Type	Extent	Role and Importance
National Renewable Energy Lab	Federal Laboratory	Major	Provided technical assistant with patented technique, full-scale web line for testing up to 100 ft/min

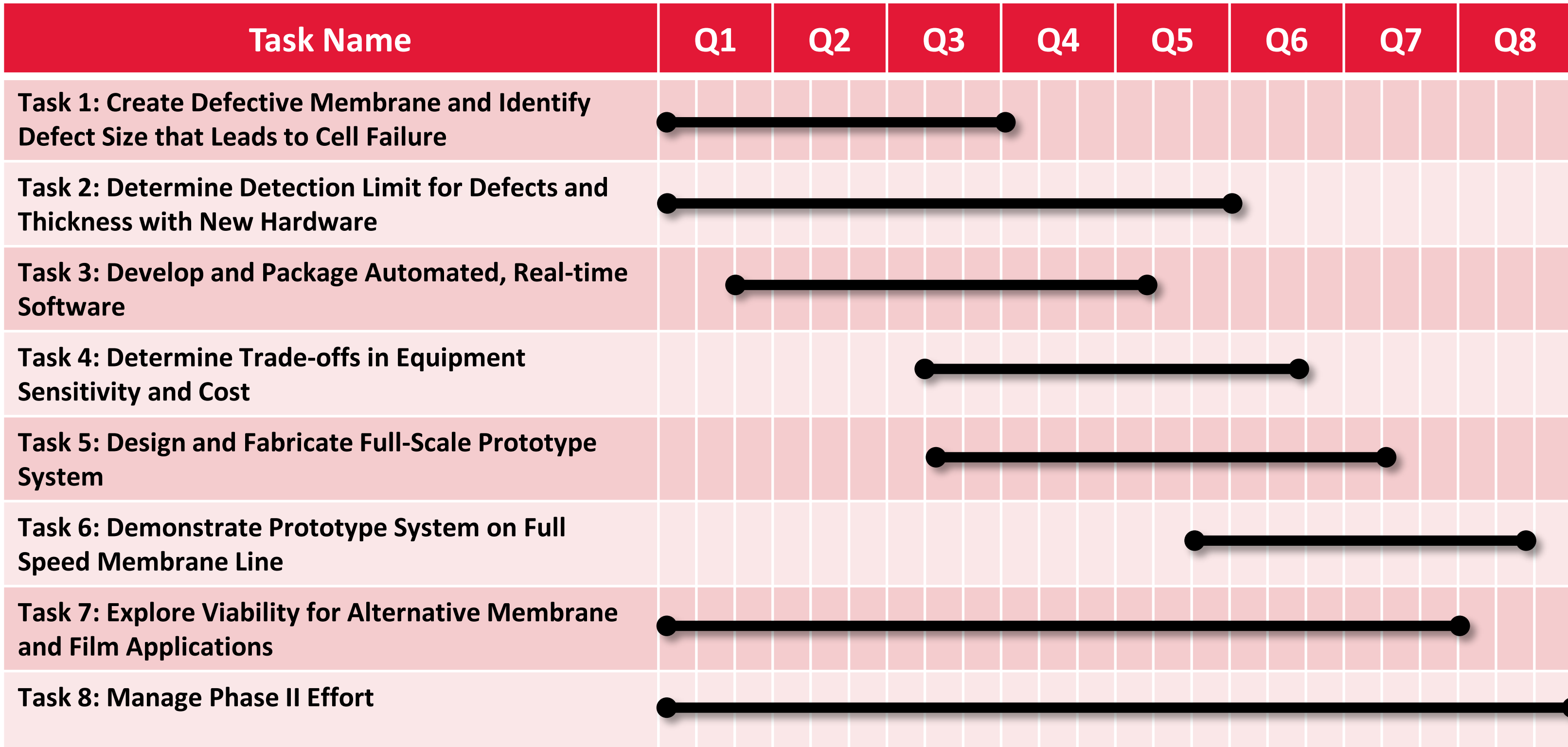
Remaining Challenges and Barriers

- **Remaining Objectives**
  - Knowledge of smallest required limit of detection
  - Testing of smallest defect with upgraded hardware
  - Full automation of software and hardware
  - Data on real web-lines
  - Trade-offs between cost and accuracy
  - Alternative membrane application testing
- **Key Barriers**
  - Access to industry web-lines
  - Testing on most relevant membranes
  - Full understanding of system requirements

Technology Transfer Activities

- Mainstream is pursuing SBIR Phase II funding to develop the system to a TRL 7 and commercialize the product
- Mainstream has an option to license two patents from NREL
- Plan to demonstrate the prototype system on two industrial web lines in addition to NREL
- While the PEM fuel cell market is the primary focus, the technology is applicable to other markets such as reverse osmosis, electrolysis, and protective films

Proposed Future Work



Proposed Future Work

- **Proposed Work**
  - Improve resolution to 4 µm incorporating high-resolution camera and high-speed processor
  - Scale system to real-time measurements of thickness over 24-inch web
  - Demonstrate reliability of packaged system for defect detection up to 100 ft/min
- **Key Milestones**
  - 4 µm defects at 100 ft/min
  - 0.5 µm thickness resolution
  - 5σ false-positive and negative rate
  - Fully packaged prototype (TRL 7)
- **Methods to Mitigate Risk**
  - Leverage NREL experience
  - Leverage expertise from other projects
  - Involve potential customers early in the development process
  - Design a low-cost variant for applications with looser tolerances
  - Explore alternative applications to broaden market and drive down cost
- **Go/No Go Decisions**
  - Full-width thickness mapping across a 24-inch web at 30 ft/min
  - Defect detection across 24-inch web at 30 ft/min

Summary

- Pinholes as small as 25 µm were successfully identified in static samples with the low cost camera system
- Demonstrated thickness mapping to a resolution of ± 1 µm for Nafion®-115 and Nafion®-211
- Demonstrated the performance of the enhanced optical techniques with 18 membranes and films including a variety of supported and unsupported membranes
- Real-time identification of 100% of 100 µm induced defects in Nafion®-211 at 30 ft/min on NREL's web line
- Defect type and position successfully logged electronically and location printed on the web

SUPPORTING SLIDES

Mainstream Engineering Corporation

- Small business incorporated in 1986
- 100+ employees
- Mechanical, chemical, electrical, materials and aerospace engineers
- 100,000 ft^2 facility in Rockledge, FL
- Laboratories: electric power, electronics, materials, nanotube, physical and analytical chemistry, thermal, fuels, internal combustion engine
- Manufacturing: 3- and 5- axis CNC and manual mills, CNC and manual lathes, grinders, sheet metal, plastic injection molding, welding and painting

Capabilities

- **Basic Research, Applied Research & Product Development**
- **Transition from Research to Production (Systems Solution)**
- **Manufacture Advanced Products**

Mission Statement

To research and develop emerging technologies.
To engineer these technologies into superior quality, military and private sector products that provide a technological advantage.

SBIR Successes and Awards

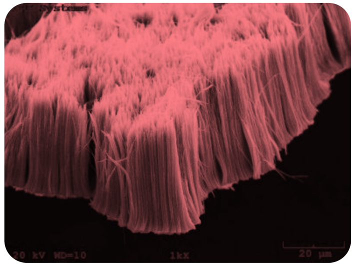
- **95% DOD Commercialization Index**
- **SBIR spinoffs – QwikProduct Line**
- **SBIR spinoffs – Military Product Line**
- **Honors**
  - 2014 DOE's SBIR/STTR Small Business of the Year
  - 2013 Florida Excellence Award by the Small Business Institute for Excellence in Commerce
  - Winner Florida Companies to Watch
  - Blue Chip Enterprise Initiative Awards
  - Job Creation Awards
  - Two SBA's Tibbetts Awards for Commercialization
  - State of Florida Governor's New Product Award
  - SBA's Small Business Prime Contractor of the Year for the Southeastern U.S.
  - SBA's Administrator's Award for Excellence

Mainstream's Focus Areas

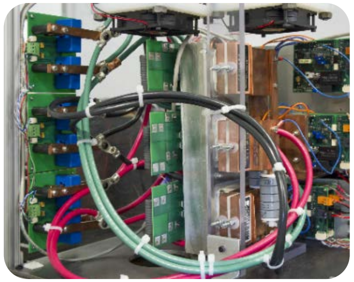
THERMAL CONTROL
- High Heat Flux Cooling
- Thermal Energy Storage
- Directed Energy Weapons
- Rugged Military Systems

ENERGY CONVERSION
- Combustion
- Diesel/JP-8 Engines
- Biomass Conversion
- Alternative Fuels
- Fuel Cells

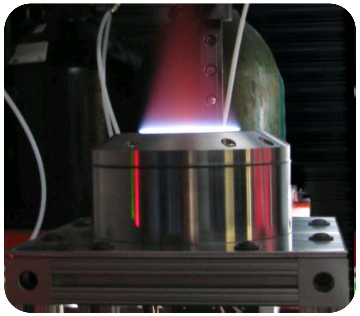
TURBOMACHINERY
- Compressors
- Turbines
- Bearings/Seals
- Airborne Power Systems



MATERIALS SCIENCE
- Thermoelectrics
- Batteries/Ultracapacitors
- Hydrogen Storage
- E-Beam Processing
- Nanostructured Materials



POWER ELECTRONICS
- High Speed Motor Drives
- Hybrid Power Systems
- Solar/Wind Electronics
- Pulse Power Supplies
- Battery Chargers



CHEMICAL TECHNOLOGIES
- Heat Transfer Fluids
- Catalysis
- Chemical Replacements
- Water Purification
- Chemical Sensors